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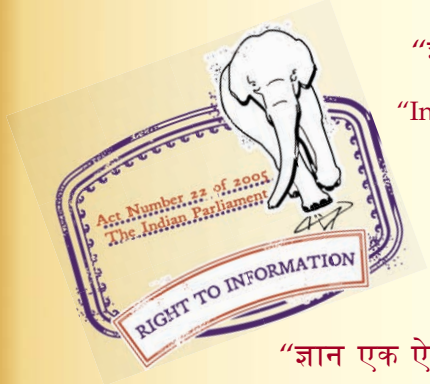
IS 7135 (1987): Dimethyl Sulphate, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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"Knowledge is such a treasure which cannot be stolen"

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IS 7135 - 1987
(Reaffirmed 2004)

Indian Standard

SPECIFICATION FOR DIMETHYL SULPHATE, TECHNICAL

(First Revision)

First Reprint FEBRUARY 1997

UDC 547.261'122.6

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 2

October 1988

Indian Standard

SPECIFICATION FOR DIMETHYL SULPHATE, TECHNICAL

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 26 November 1987, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Dimethyl sulphate [$(CH_3)_2 SO_4$] is widely used in organic synthesis as methylating agent. Dimethyl sulphate is poisonous and hence sufficient care is required to be taken in handling the material (*see* **3** and Note 2 under **A-1.1.1**).

0.3 This standard was first published in 1973. The Committee decided to revise it in view of the experience gained over the past. In the present version, the details of test method for the requirement of free acidity (as H_2SO_4) have been modified.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for dimethyl sulphate, technical.

2. REQUIREMENTS

2.1 Description — The material shall be transparent liquid and colourless to pale-yellow in colour. It shall be miscible with ethyl alcohol and acetone. It shall also be free from suspended matter.

2.1.1 The material is slowly decomposed by water. It also darkens on keeping.

2.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods given in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

3. PRECAUTIONS IN HANDLING AND STORING

3.1 The vapours of dimethyl sulphate are powerful irritant to mucous memberanes and produce severe toxic effects. Extreme care, therefore, shall be taken at all stages of handling and storage.

TABLE 1 REQUIREMENTS FOR DIMETHYL SULPHATE, TECHNICAL

(*Clauses* 2.2 *and* B-4.1)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Appendix A)
(1)	(2)	(3)	(4)
i)	Relative density at 27°C/27°C	1·318 to 1·330	A-3
ii)	Dimethyl sulphate content, percent by mass, *Min*	98·5	A-4
iii)	Free acidity (as H_2SO_4), percent by mass, *Max*	0 50	A-5

4. PACKING AND MARKING

4.1 Packing

4.1.1 The material shall be supplied, in well-closed glass carboys or any other suitable containers as agreed to between the purchaser and the supplier.

4.1.2 The material shall be stored in a cool place.

4.2 Marking

4.2.1 The container shall be marked with the following information apart from the minimum cautionary notice worded as follows:

'WARNING — POISONOUS'

a) Name of the material;

b) Manufacturer's name and recognized trade-mark, if any;

c) Net mass of the material; and

d) Lot or batch number, in code or otherwise.

4.2.1.1 The container shall also be clearly labelled as in Fig. 5 of IS : 1260 (Part 1)-1973*.

4.2.1.2 The top side of the containers shall also be indicated.

*Pictorial markings for handling and labelling of goods: Part 1 Dangerous goods (*first revision*).

4.2.2 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 Representative samples of the material shall be drawn and their conformity to the requirements of this standard shall be judged as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.2 and *Table* 1)

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in the tests.

NOTE 1 — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

NOTE 2 — Since the vapours of dimethyl sulphate are powerful irritant to mucous membranes, it should be very carefully handled observing the information given in the Chemical Safety Sheet SD 19, published by the Manufacturing Chemists' Association, Inc.

A-2. SAMPLES FOR TEST

A-2.1 Mix the sample well by rotating the bottle several times and transfer a portion immediately into another bottle and stopper it. Do not expose the sample to an atmosphere containing acid or alkaline fumes.

A-3. DETERMINATION OF RELATIVE DENSITY

A-3.0 Outline of the Method — In this method, masses of equal volumes of the material and water at the same temperature are compared using pyknometer or relative density bottle.

A-3.1 Apparatus

A-3.1.1 *Pyknometer* — 25-ml capacity (*see* IS : 5717-1970†).

*Specification for water for general laboratory use (*second revision*).

†Specification for pyknometers.

A-3.1.2 *Water-Bath* — maintained at 27·0 ± 0·2°C.

A-3.2 Procedure — Weigh a clean, dry pyknometer. Fill the tared pyknometer with freshly boiled distilled water and place it in a bath maintained at 27·0 ± 0·2°C and allow sufficient time (about 30 minutes) to attain the temperature of the bath. Then insert the capillary stopper which has also been brought to 27·0 ± 0·2°C. Wipe the excess liquid from the stopper, remove the pyknometer from the bath, bring room temperature and weigh. Empty the pyknometer, clean and dry it, and repeat the operation with material at 27·0 ± 0·2°C.

A-3.3 Calculation

$$\text{Relative density at } 27°C/27°C = \frac{M_1 - M_2}{M_3 - M_2}$$

where

M_1 = mass in g of the pyknometer filled with the material,

M_2 = mass in g of the dry pyknometer, and

M_3 = mass in g of the pyknometer filled with water.

A-4. DETERMINATION OF DIMETHYL SULPHATE CONTENT

A-4.0 Outline of the Method — Dimethyl sulphate on hydrolysis yields methyl sulphuric acid and methanol. Methyl sulphuric acid is then titrated against standard sodium hydroxide solution to determine dimethyl sulphate content.

A-4.1 Reagents

A-4.1.1 *Sodium Hydroxide Solution* — approximately 0·5 N.

A-4.1.2 *Standard Hydrochloric Acid* — 0·5 N.

A-4.1.3 *Phenolphthalein Indicator* — 0·5 percent (*m/v*). Dissolve 0·5 g of phenolphthalein in rectified spirit (*see* IS : 323-1959*) and make it faintly pink with dilute sodium hydroxide.

A-4.2 Procedure — Take about 0·5 g of the material accurately weighed in a 250-ml stoppered flask, add 25 ml of sodium hydroxide solution and allow it to stand for 2 hours at room temperature with occasional shaking. Titrate the excess of alkali with standard hydrochloric acid using phenolphthalein as indicator. Carry out a blank with 25 ml of sodium hydroxide solution.

A-4.3 Calculation

$$\text{Dimethyl sulphate content, percent by mass} = \frac{12{\cdot}614\,(V - V_1)\,N}{M} - A$$

where

V = volume in ml of standard hydrochloric acid used in the blank determination,

V_1 = volume in ml of standard hydrochloric acid consumed in the test with the material under test,

N = normality of standard hydrochloric acid,

M = mass in g of the material taken for the test, and

A = dimethyl sulphate content equivalent to the acidity (*see* **A-5**). This value is equal to $\frac{126}{98}$ × acidity.

A-5. DETERMINATION OF FREE ACIDITY

A-5.0 Two methods, namely, Method *A* and Method *B*, have been prescribed. Method *B* shall be used to routine analysis whereas Method *A* shall be used as referee method.

A-5.1 Method *A*

A-5.1.1 *Reagents*

A-5.1.1.1 *Standard sodium hydroxide solution* — 0·1 N.

A-5.1.1.2 *Phenolphthalein indicator* — same as in **A-4.1.3**.

A-5.1.1.3 *Solvent ether*

A-5.1.2 *Procedure* — Transfer exactly 10 ml of the material to a dry separating funnel of 250-ml capacity. Add about 100 ml chilled distilled water (0 to 5°C) in the separating funnel. Swirl for 60 seconds and immediately separate the dimethyl sulphate layer. Remove the dimethyl sulphate layer and extract the aqueous layer with 50 ml solvent ether. Remove the etherial layer. Repeat the extraction with another 50 ml solvent ether.

Titrate the aqueous layer with standard sodium hydroxide solution using phenolphthalein as indicator to the first appearance of pink colour by gentle shaking.

A-5.1.3 *Calculation*

$$\text{Free acidity (as } H_2SO_4\text{), percent by mass} = \frac{4{\cdot}904\,V \times N}{V_2 \times d}$$

where

V = volume in ml of standard sodium-hydroxide solution used in the titration.

N = normality of the standard sodium hydroxide solution,

V_2 = volume in ml of the material, and

d = relative density of the material.

A-5.2 Method *B*

A-5.2.1 *Reagents*

A-5.2.1.1 *Alcohol* — 95 percent.

A-5.2.1.2 *Standard alcoholic sodium hydroxide solution* — 0·1 N.

A-5.2.1.3 *Phenolphthalein indicator solution* — Dissolve 0·5 g of phenolphthalein in 100 ml of rectified spirit (*see* IS : 323-1959*) which has previously been neutralized to the indicator.

A-5.2.2 *Procedure* — Add 10 ml of the material to about 50 ml of previously neutralized alcohol contained in a 250-ml conical flask.

Titrate the contents with standard alcoholic sodium hydroxide solution using phenolphthalein as indicator to the first appearance of pink colour by gentle shaking.

A-5.2.3 *Calculation*

$$\text{Free acidity (as } H_2SO_4\text{), percent by mass} = \frac{4{\cdot}904\,V \times N}{V_2 \times d}$$

where

V = volume in ml of standard alcoholic sodium hydroxide solution used, and

N, V_2 and d have the same significances as in **A-5.1.3**.

*Specification for rectified spirit (*revised*).

*Specification for rectified spirit (*revised*).

APPENDIX B

(*Clause* 5.1)

SAMPLING OF DIMETHYL SULPHATE, TECHNICAL

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw representative sample, the contents of each containers selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers on which the material has no action.

B-1.6 The sample container shall be of such a size that they are almost three-fourths filled by the sample.

B-1.7 Each sample container shall be sealed air-tight after filling, and marked with full details of sampling and the date of sampling.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the same size containing material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute a separate lot.

B-2.2 The number of containers (n) to be selected from a lot shall depend on the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FROM LOTS OF DIFFERENT SIZES

(*Clause* 6.2.2)

Lot Size N	Sample Size n
(1)	(2)
Up to 15	3
16 ,, 40	4
41 ,, 65	5
66 ,, 110	7
111 and above	10

B-2.3 The containers shall be selected at random from the lot and in order to ensure random sampling, methods given in IS : 4905-1968* may be followed.

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 From each of the containers selected according to **B-2.3**, a representative portion of the material, about 300 ml in volume, shall be drawn. From each of these individual portions, an equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample not less than 600 ml in volume. The composite sample shall be transferred to clean bottles and labelled with full identification particulars of the sample. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-4. NUMBER OF TESTS

B-4.1 Tests for determination of characteristics given in Table 1 shall be carried out on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 The lot shall be declared as conforming to this specification if all the test results on the composite sample satisfy the corresponding requirements specified in this standard.

*Methods for random sampling.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at New India Printing Press, Khurja, India